

CFUR # 0509-07-100 9.27

Preparation of 1168R-H2O

FROM EURACA # 06 by 1st method

# RAW MATERIALS

EURACA #06 (wet) 116.8g or 1050.0g

DMW 443.2g ml

THE 500.0g

MAEM 74.0g

TEA 40.0ml

ETOAC (750 + 400) ml

DMW 200.0ml

NaCl 135.0g

THE (950 + 100) ml

Carbon 15.0g

cor. HCl (21.0 + 19.5 + 12.0) ml

IPE 450.0 ml

IPE (washing) 250.0 ml

EDTA 1.0g

[500 - (wet wt - 60)]

## PROCEDURE

1. charge DMW & THE then cool to 5°C
2. charge wet EURACA followed by MAEM at 3-5°C
3. Add TEA in 3hrs at 3-5°C and maintain the temp at 3-5°C
4. upto complete reaction
5. charge ETOAC 750 ml and EDTA 1.0g at 15°C for 15 min. separate the layers
6. Extract the org. layer with 400 ml ETOAC for 15' & separate the layers. (settle it for 15')
7. Extract the mixed ETOAC layer (10.0g = 1.0 ml)

- 200 ml DMW and then  
 add this aq. soln into rich aq phase  
 meth 7 To this aq. soln add NaCl &  
 950 ml THF at 18-20°C  
 8 Adjust the pH to 3.0-3.1 & con Hcl  
 at 18-20°C in 20-25'  
 9 Separate the layers & eliminate  
 the aq phase  
 10 charcoalise the org phase & for 40'  
 at 18-20°C  
 11 Filter it & wash the bed with 100 ml THF  
 12 Adjust the pH to 1.0-0.9 by  
 con Hcl in 10-15' at 18-20°C  
 13 Seed with 1.0 g CFUR Hcl and  
 stir for 1 hr at 18-20°C  
 14 Again bring the pH to 1.0-0.9  
 by con Hcl at 18-20°C  
 15 Seed again with 1.0 g CFUR Hcl &  
 stir for 1 hr at 18-20°C  
 16 Add 450 ml IPE in 40-45' at 18-20°C  
 17 Stir for 1 hr at 18-20°C & filter it  
 18 Wash the product with 250 ml IPE  
 19 Dry the product UV at 40°C up to 10 mg  
 MC < 5.0%

DURATION Rxn Monitoring RESULTS

|       | EDRACA | CFUR  | MAEM | CFT  | MBT   |
|-------|--------|-------|------|------|-------|
| 18hr. | 2.43   | 76.33 | 5.97 | 0.38 | 12.74 |

Terminated in 18<sup>1</sup> hr  
18hr.

FOR

FLC  
2996

## OBSERVATION:

1. Reaction mixture is clear sol<sup>n</sup> after 10 hr. stirring but very dark in colour
2. Vol. of Ag<sup>+</sup> sol<sup>n</sup> is 940.0 ml
3. Vol. of EtOAc layer is 1570.0 ml
4. pH of Ag<sup>+</sup> sol<sup>n</sup> + NaCl + THF is 8.30
5. Vol. of CFUR Rich THF layer is 1135.0 ml (B.C)
6. Vol. of Ag<sup>+</sup> sol<sup>n</sup> containing NaCl is 790.0 ml
7. After 30% characterization, colour of the rich sol<sup>n</sup> is lighter like STD.
8. Only 0.5g CFUR HCl is seeded in the stirring abundant amount of material comes out.
9. Colour of slurry is better than #03 & 04.
10. Colour of dried product is slightly better than #03 & 04.

| Dry wt | CFUR Assay | CFT  | Imp. | FURAC. m/c         |
|--------|------------|------|------|--------------------|
| 87.4g  | 97.45      | 0.59 | 0.37 | 4.31               |
|        |            |      |      | 0.10 (qualitative) |

CV: 0.27%